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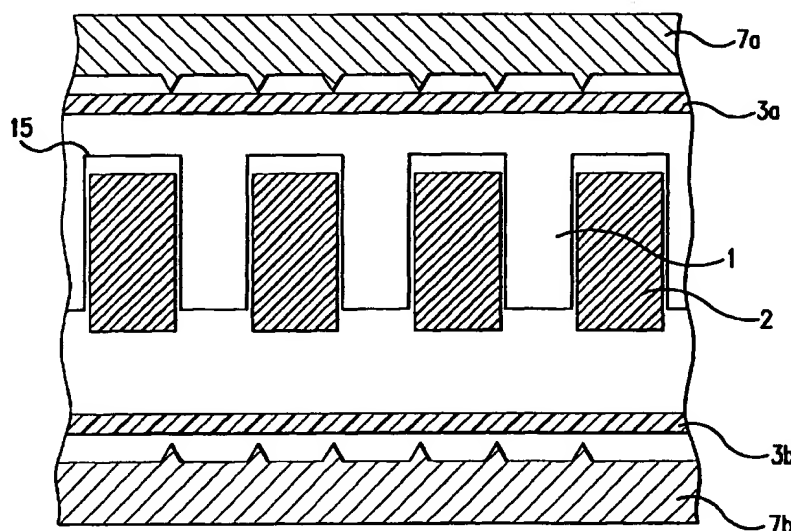
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- (71) Applicant (for all designated States except US): UOP LLC [US/US]; 25 East Algonquin Road, Des Plaines, IL 60017-5017 (US).
- (72) Inventors; and
- (75) Inventors/Applicants (for US only): AKPORIAYE, Duncan, E. [GB/NO]; Sintef, 124 Blindern, N-0314 Oslo (NO). DAHL, Ivar, M. [NO/NO]; Sintef, 124 Blindern, N-0314 Oslo (NO). KARLSSON, Arne [SE/NO]; Sintef, 124 Blindern, N-0314 Oslo (NO). WENDELBO, Rune [NO/NO]; Sintef, 124 Blindern, N-0314 Oslo (NO).
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(54) Title: MULTIAUTOClave WITH LINER FOR COMBINATORIAL SYNTHESIS OF ZEOLITES AND OTHER MATERIALS



(57) Abstract: The invention relates to a multiautoclave and details of its design. The multiautoclave consists typically of a pressure vessel/autoclave with from 10 to 10,000 small, separated chambers, each typically having a volume of from 0.2 to 2 mL. The chambers are preferably formed from through-going perforations in a central block with the perforations being sealed by means of balls, septa, stoppers, Teflon™ sheets or such which are placed at the bottom and top of each through-going perforation. The perforations are lined with a liner of inert material that extends into each of the perforations. Metal plates are placed over and under the central block so that the locking mechanism presses the individual parts and the seals together with sufficient load to enable the chambers to be filled with liquid and to be heated without the occurrence of leakage.

WO 02/07873 A2



*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

# "MULTIAUTOCLAVE WITH LINER FOR COMBINATORIAL SYNTHESIS OF ZEOLITES AND OTHER MATERIALS"

## FIELD OF THE INVENTION

The present invention relates to a pressure and temperature reactor  
5 vessel having a liner and particularly to a lined multiautoclave.

## BACKGROUND OF THE INVENTION

Many materials, such as, e.g., zeolites, are prepared by so-called  
hydrothermal synthesis at temperatures ranging from 100°C to 200°C requiring  
crystallization times of one hour or more. For syntheses being carried out at  
10 temperatures that are higher than the boiling point of the solvent, it is necessary  
to use pressure vessels, and these have to be suitable for the temperature and  
pressure used during the operation. The pressure vessel should be designed so  
that the handling of the pressure vessel does not present any unnecessary  
hazard, provided it is used according to working instructions.

15 Zeolite syntheses are usually performed in strongly alkaline media, often  
at pH>14, and the reaction mixture will often contain toxic chemicals such as,  
e.g., fluoride. Conventionally, syntheses that may be performed at temperatures  
lower than 110°C are carried out in polymer bottles, often Teflon™, while  
reactions at higher temperatures require steel autoclaves, perhaps lined with  
20 Teflon™. The price of an autoclave of this type with the required safety details is  
typically of the order of about 1,000 United States dollars (about NOK 10,000) or  
higher. Furthermore, such an autoclave will weigh from 1 kilogram and upwards,  
and all of these elements represent limitations regarding the number of  
syntheses that may be performed in most laboratories in the course of one year.

25 Zeolite synthesis is often carried out by keeping the synthesis mixture at  
around 100°C for at least 6 h. At these moderate temperatures sealed chambers  
are necessary in order to avoid drying out of the synthesis mixture. As an  
example of conventional zeolite synthesis, Zeolite Y can be prepared according  
to US-A-3130007, Example 1, by dissolving 5 g sodium aluminate containing 30  
weight percent Na<sub>2</sub>O and 44 weight percent Al<sub>2</sub>O<sub>3</sub> and 22 g sodium hydroxide  
30 containing 77.5 weight percent Na<sub>2</sub>O in 89,5 ml distilled water. This solution was  
added to 124.2 g of an aqueous colloidal silica sol with 29.5 weight percent SiO<sub>2</sub>,

so that the resulting mixture had a composition corresponding to 13.9 Na<sub>2</sub>O : Al<sub>2</sub>O<sub>3</sub> : 28.2 SiO<sub>2</sub> : 471 H<sub>2</sub>O, and the mixture was homogenized by stirring. The mixture was enclosed in a sealed glass vessel, placed in a water bath and heated at 100°C for 21 hours, after which the product was recovered by filtering, washed and dried. Common to all the synthesis procedures mentioned and for all other known synthesis procedures for the preparation of zeolites on a laboratory scale with the purpose of discovering new zeolites or to optimize existing zeolites is that these are performed in a cumbersome and expensive manner by having to separately prepare each reaction mixture, which typically consists of 4-7 reagents, and by adding the reagents one by one.

In many other examples the synthesis of zeolites and other molsieves needs temperatures well above 100°C, so that steel pressure vessels or the like are required. Furthermore, each reaction mixture is typically prepared in batches of 5 to 100 g and crystallized in expensive and heavy autoclaves with internal volumes often in the range of 25 to 250 ml and with weights of up to 8 kg per autoclave, causing considerable expense due to a large consumption of often expensive reagents and due to the fact that the handling of the heavy autoclaves often makes it impossible to handle more than one autoclave at a time, and finally that the size of the autoclave limits the number of autoclaves that may be placed in each oven or heating unit. The combination of all of these elements is, according to known technology, making each zeolite synthesis a very resource intensive process, and there is a great need for greater efficiency, rationalization, downscaling and automation. Simple calculations have shown that by combining the different variables which are involved in zeolite synthesis with narrow enough intervals in reagent concentrations, temperatures, reaction time, etc. to cover any phase formation based on known examples, it is feasible to make up 10<sup>18</sup> recipes. With today's synthesis capacity, which on a global basis hardly exceeds 100,000 synthesis per annum, it would take 10,000,000,000,000 years to carry out all these syntheses in which each and every one in theory has potential for the preparation of a new zeolite or other microporous material. The expenses involved in performing these syntheses according to known technology would obviously be formidable, and there is thus a great need for new and more cost efficient methods for zeolite synthesis.

In recent years, new, automated methods for the systematic preparation of new compounds, so-called "combinatorial techniques," have been developed, but equipment which may be used for liquid phase synthesis at temperatures above approximately 100°C has until now not been disclosed, because this temperature requires the synthesis to be performed in a hermetically sealed vessel at elevated pressures. WO 95/12608-A1, for instance, discloses an apparatus and a method for a) synthesis of several molecules on substrates, comprising distribution of the substrates in the reaction chambers, b) combination of the first addition of these molecules with different reagents in each of the reaction chambers, c) moving the substrates through tubing to separate mixing chambers where the substrates are mixed, d) redistribution of the substrates by transport through tubing back to the reaction chambers, and e) combination of a second portion of different composition to the first portions of molecules in the different reaction chambers in order to prepare new mixtures. This publication describes only a system for mixing and distribution of different molecules and not a system for hermetical sealing of the reaction chambers which would make it possible to operate at high temperatures, and this system would thus not be suitable for the synthesis of zeolites. In WO 96/11878 there is a description of extensive use of a combinatorial arrangement for synthesis of new materials, including zeolite synthesis at 100°C. Even though this reference presents a detailed description of instrumentation and equipment developed for different purposes, autoclave systems required for performing the syntheses under the prevailing physical conditions (elevated pressure and temperatures exceeding 100°C) are not described.

Until recently there has been no available literature describing equipment that might be used for practical work with combinatorial zeolite synthesis, inasmuch as such syntheses almost without exception require hydrothermal treatment of a solution or gel with a relatively high content of water and often high contents of organic compounds in a closed chamber, and almost all methods for preparation of zeolites known thus far require such conditions during synthesis. This is true without exception for all methods which have proven to be commercially applicable. The synthesis of zeolites is thus normally performed under hydrothermal conditions which require elevated pressures and high

temperatures in periods up to several weeks without leakage. The problem has been the costs involved in this type of work, estimated to an average of about 500 to 600 United States dollars (about NOK 5,000) per synthesis, including recovery of the product and XRD analysis. An important feature when dealing with large series of syntheses is therefore how the product can be recovered and washed in a convenient manner without insurmountable expense.

WO 98/36826 discloses a system for screening of synthesis conditions for the preparation of zeolites and other non-carbon materials requiring hydrothermal conditions in the temperature range of 100°C to 250°C in a cost efficient manner. Some of the parameters that have been made more cost efficient with the multiautoclave of WO/9836826 include: reduced size of the separate reaction chambers and increased number of reaction chambers in what is known as a multiautoclave; reduced use of reactants and thus cheaper synthesis; automated addition of reactants, for instance by having 100 reaction chambers present in one multiautoclave and by enabling this to be connected to a pipetting machine which makes quick and exact addition of all liquid reactants possible; simple and easy-to-use mechanism for the closing and opening of the multiautoclave; simple recovery and washing of the synthesis product and simple cleaning of the multiautoclave after use; and devices allowing automated analysis with X-ray diffraction and automatic identification of known crystalline phases by combination of an automatic sample switcher, a structure library stored in a database and software that can monitor sample switching and identification. WO/9836826 has also disclosed automated equipment for larger synthesis series and preparation formulations based on mixtures of different liquids/solutions with varying reactant ratios.

The invention of WO/9836826 represents a breakthrough in terms of cost reduction for zeolite synthesis in that the reaction mixture crystallizes in a volume reduced typically to 1/100 of what has been used conventionally, thereby achieving reduced consumption of reactants and cheaper syntheses, and further by enabling automated addition of reactants, e.g., by having 100 or more available reaction chambers in one single multiautoclave, and that the multiautoclave plates can be connected to a pipetting machine that makes quick and exact addition of all liquid reactants possible, and by being able to place

several such plates with reaction chambers on top of each other without difficulty. Furthermore, an important feature of the WO/9836826 invention is the simple and not very time-consuming operation of the multiautoclave.

The WO/9836826 invention relates to a pressure and temperature reactor  
5 vessel comprising a central block having a multitude of perforations, wherein said perforations are through-going perforations, or cavities or other form of holes permanently closed at one end, a cover means, operatively associated with a sealing means, for engagement with said central block to seal the open  
10 ends of said perforations forming a multitude of chambers, a sealing means, operatively associated with the cover means, to form a pressure tight seal when said cover means is brought into position by a locking means, and a locking means acting in concert with the cover means to engage the sealing means so as to define a multitude of reaction chambers. Applications for the WO/9836826 invention may, in addition to zeolite synthesis, be in any field of activities within  
15 research and development connected to products where at least one production step comprises the mixing of different liquids, e.g., in the fields of organic and inorganic syntheses, paint production, formulation of fuels, food industry, etc., and, furthermore, applications within clinical testing, dissolution and digestion of samples with acid etc. where a liquid reactant is added to a liquid or solid. The  
20 invention of WO/9836826 is, in particular, aimed at applications where open vessels cannot be used, and more specifically for applications where it is required to operate at temperatures which will cause elevated pressures in the liquid part of the mixture.

The present invention is an advancement of the invention disclosed in  
25 WO/9836826 in that a liner is used within the multiautoclave. The liner is constructed of material that is inert in the reaction conducted within the multiautoclave, and the liner is preferably thin. The liner allows for a simple means of extracting solid material from the multiautoclave and can then be replaced with a fresh liner to minimize cross contamination between runs using  
30 the multiautoclave. Optionally, the liner may be used in the weighing of reagents such as powders and liquids for increased accuracy. Others have employed a liner in specific single vessel units such as US-A-4554136 where a fluoropolymer lining is used to inhibit acid corrosion of the walls of the pressure vessel, US-A-

3048481 which discloses a refractory lining used within a synthesis gas generator, and US-A-3396865 which teaches a synthesis pressure vessel having a thermally conductive pressure shell and a chemically resistant thermally insulating lining within the shell made of a dense refractory concrete. The present invention, however, is unique in its use of a liner to facilitate solid product removal and minimize cross contamination between runs using the multiautoclave.

### SUMMARY OF THE INVENTION

The invention comprises a unit containing a multitude of pressure vessels, also referred to as a multiautoclave. The multiautoclave has typically from 10 to 10,000 or more small, separate chambers (1), each typically with a volume of 0.001 – 10 ml. The multiautoclave is composed of a central block and a set of plates (2 and 7) having protrusions (4) a thin liner (15) lining the small separate chambers and optionally thin laminate (3a and 3b) which are stacked so that they form a matrix of small chambers (1), as shown in Figures 1 – 5. The central block consists of a perforated plate (2), with a multitude of perforations, which, when sandwiched between the plates (7a and 7b) and closed, serve as the frame of the pressure chambers.

### BRIEF DESCRIPTION OF THE DRAWINGS

Further advantages and characteristic features of the present invention are illustrated in the following description by referring to the drawings which, as mere examples without limitations, show some designs related to this invention, where Figure 1 presents a side view of the multiautoclave with the single components disassembled, and Figure 2 shows a top view of the multiautoclave. Figure 3 shows the side view of an alternative design of the multiautoclave which employs septa between the plates. Figure 4 depicts the side view of yet another design of a multiautoclave where the perforated plate has protrusions at the edges of the perforations to engage the seals. Figure 5 illustrates the side view of a multiautoclave where the plates have protrusions to engage the seals. Figure 6 shows a multiautoclave design having a single plate and partial (non-through-going) perforations of the central block.



## DETAILED DESCRIPTION OF THE INVENTION

As summarized above, the invention is an apparatus containing a multitude of pressure vessels, also referred to as a multiautoclave. The small separate chambers that define the pressure vessels are made up of a set of  
5 plates, an optional thin laminate, a central block, sealing means, and a liner. The individual components are stacked so that they form a matrix of small chambers, as shown in Figures 1 – 6. The thickness of the different plates and laminae may vary, always keeping in mind that a maximum number of small chambers (1) with optimal size is desired, and that the chambers must be leak tight and  
10 dimensioned such that no excessive degree of deformation will take place under the operational conditions for which the equipment is designed.

The central block consists of a perforated plate (2), with a multitude of perforations and when sandwiched between the plates (7a and 7b) and closed, serve as the frame of the pressure chambers. When the apparatus is to be used  
15 at temperatures in the range of from 150 to 250°C, the central block may be made from stainless steel, aluminum, titanium or other rigid material such as polyethylethylketone (PEEK) or the like, with perforations of, e.g., 10 mm diameters. For use at temperatures above 150°C, the central block can be made entirely of Teflon™, for use below 130°C it can be made of polypropylene, and  
20 for use below 105°C it can be made of polyethylene. It is preferred that the perforations are through going, or in other words, the perforations extend from one surface of the central block to a parallel second surface of the central block. Although less preferred, the perforations may define cavities that do not extend completely through the central block, see Figure 6. In general, the invention will  
25 be described below as the preferred embodiment of having through-going perforations.

A thin liner (15) that preferably conforms to the shape and perforations of the central block is positioned so that the liner extends into each of the perforations. The liner lines the walls of the perforations and lines or forms a  
30 bottom to the perforations, see Figure 1. The liner is preferably made of an inert polymer material, such as Teflon™, polyethylene, polypropylene, perfluoroalcoxy fluorinated ethylene propylene, and polyethylethylketone, that is able to  
withstand the temperatures and pressures necessary for synthetic reactions. It is

also preferred that the liner be thin and lightweight. A preferred range of thickness for the liner is from 0.1 to 1 mm. The liner provides several advantages with the most important being the simple means of extracting solid products from the separate reaction chambers or perforations. Another advantage is the significantly reduced chance of cross contamination between runs using the multiple pressure vessels. The liner may also be used to weigh the reagents to a high degree of accuracy. Due to the liner being thin, the reduction in the volume of the reaction chamber is not significant. The liner may also provide an alternative approach to product recovery when the central block contains through-going perforations, specifically, the synthesis products may be pressed out of the perforation by inverting the liner wells. This embodiment is an option when the liner is sufficiently thin to allow the bottom of the portion of the liner that is extended into the perforations to be pressed through the block.

It is preferred that bottom and top plates (7a and 7b) are designed with structures that have conical or hemispheric protrusions (4) that fit into the perforations. It is also preferred to have polymer films molded or otherwise shaped to cover all the perforations. When the plates are assembled with the central block, the protrusions fit into the openings of the small chambers and along with the polymer films provide a seal.

In another less preferred design, the central block has been machined so that sharp edges (14) protrude around each perforation or well, as shown in Figure 4. The advantage of this design is that one smooth polymer sheet can be used as a lid or sealing device for all the chambers because the sharp protrusions cut into this polymer sheet so that leakage between adjacent chambers does not occur when the reaction vessel is closed with its locking mechanism. The sharp protrusions (14) could alternatively consist of rings or, e.g., a rectangular grid that are either welded onto the central block (2) or fastened by any other suitable method. These protrusions may optionally be part of the insides of the top (7a) and bottom plates (7b), as shown in Figure 5 or the central block (2) as shown in Figure 4. The important point here is that the load used when assembling the different elements is concentrated just at the edges of the perforations and lids, or another appropriate contact point, so that liquid or vapor cannot leak out during heating.

Less preferred embodiments include using, in the separate pressure chambers, balls made from Teflon™ or other suitable material such as steel, PEEK, nylon or glass as seals for use in conjunction with the plates to seal the perforations. Other sealing means or septa (5) made from an elastomer or from another appropriate material such as Teflon™ in the shape of circular disks will give an advantage in the form of reduced weight and volume. Septa should be made from an elastomer, preferably Viton, or other suitable material that can withstand temperatures of at least 200°C, and they may be equipped with Teflon™ lining (13) on the side facing the chamber as shown in Figure 3. Furthermore, septa (5) should have a thickness considerably larger than the depth of immersion, as shown on Figure 3, in order to ensure a tight fit when assembling the different elements. The septa or balls may be fastened on the bottom and top plates or on separate polymer films or a thin metal plate in such a way that they may be put in place and removed in a simple manner.

In one embodiment, all plates and laminae have a series of holes drilled through (6) for locking means, which can be bolts which are used to assemble the multiautoclave and ensure a sufficient counter-pressure to prevent leakage when the liquid in the chambers is heated to high temperatures, and the bolts (11) and nuts (12) are placed in such a manner and their number is adjusted so that a sufficiently distributed even load is obtained in order to ensure that all the chambers are tight when in use. The multiautoclave may be closed by pressing the plates against each other employing a clamping or squeezing mechanism that makes through-going bolts unnecessary. The squeezing mechanism may include springs or the like, which ensures the maintenance of a suitable pressure. A frame made of a rigid material that ensures good tightness in the outer chambers may enclose the entire multiautoclave, also counteracting deformation of plates made of pure Teflon™ or another ductile material. Bolts and frames or whatever other locking mechanisms used are tightened with a torque wrench in order to ensure correct load throughout. A possible feature of the design is that a large number of plates may be placed on top of each other forming layers of reaction chambers according to the desired capacity. As an example, 10 plates with pressure chambers can be placed on top of each other

without requiring a total multiautoclave thickness of more than typically 25 – 40 cm.

Another important feature of the invention is that the large number of chambers which are created by the described assembly of the various described parts are lined with an inert liner. This makes simple and efficient recovery of the synthesis products possible. For example, the top plate with the associated film is removed first, and then the liner is lifted out of the central block. Each of the individual sets of reaction products corresponding to each reaction chamber is removed from the central block without cross contamination between the chambers. Also, the synthesis products remain in the same relative position as they were in the perforations of the central block in the multiautoclave, so that identification of the synthesis products can be easily maintained. The samples are retained in the well-defined matrix that, in principle, in a simple manner can be transferred to an automatic sample-switching unit for analysis, e.g., by X-ray diffraction. It is often desirable to calcine inorganic samples after synthesis, and for this purpose the samples can be washed from the liner and into the perforations of another block which is made of a material suitable for performing calcination such as stainless steel or quartz. An added advantage of using the liner is that the central block is ready to be used again with no or only minimal cleaning. Residue from the previous reactions is removed in the liner and the perforations of the central block are virtually residue-free for subsequent synthesis reactions.

Whenever the perforations, which on closing form the pressure chambers, are going entirely through the central block, the bottom portion of the liner serves to close one end of the perforation before charging with liquid or other reactants. To add support, one end of the perforation may also be physically closed by placing the perforated block (2) on top of a plate (7b). The plate and the block (2 and 7b) may then be bolted together with a set of bolts with lengths somewhat less than the combined thickness of the two plates, so that no part of the bolts is protruding from the plate assembly. After the chambers have been charged, the top plate (7a) with its sealing devices is put in place, and another set of bolts (11), which are considerably longer than the thickness of the entire multiautoclave, is put through a separate set of through-going holes (6) and

tightened with nuts (12) at the bottom side with a torque wrench and with a load sufficient to keep the multiautoclave tight under the prevailing synthesis conditions to which it will be exposed. Springs adjusted to a suitable pressure can, for instance, be put on the bolts before placing the nuts on them.

5       The overall benefits of the advances in the present invention are primarily related to the increase in efficiency in removing the synthesized products, the reduction in cross contamination, and the increase in efficiency in preparing the apparatus for subsequent use. Advances in the automated layout will make it possible to more efficiently perform large numbers of syntheses/formulations  
10 simultaneously, and it will thus be very useful for all research laboratories in industry as well as in research institutions/universities.

      Multiautoclaves suitable for the purposes mentioned above might be designed as described in the following examples, but the descriptions are to be considered merely as examples of possible designs and the given  
15 measurements and other details shall not be considered to be limitations to the invention. One of ordinary skill in the art would readily understand the invention as applied to, for example, a central block where the perforations are not through-going, and where a single plate is employed, as in Figure 6.

#### Example 1

20       A multiautoclave was built from 6 layers as shown in Figure 1. The different layers were placed on top of each other and bolted together so that multiple hermetically sealed chambers (1) are formed. By stacking several layers in a suitable way, it would be possible to make a multiautoclave with, e.g., from 100 to 1000 chambers or more. Decisive elements in the invention described  
25 here are the combination of hemisphere protrusions (4) and a thin polymer film (3) for the closing of the small chambers in the multiautoclave and the liner (15). Inasmuch as the contact between the edge of the perforation the film and the liner with the hemisphere protrusion is exposed to the total pressure developed by tightening the bolts, the multiautoclave chambers are further tightened by the  
30 slight deformation of the ductile parts.

      The multiautoclave was, from bottom to top, built from the following elements as shown in Figure 1. Bottom plate (7b) made from aluminum or steel,

with multiple symmetrically positioned hemisphere protrusions (4), each with a diameter of 13 mm. In addition, the plate had nine smaller, through-going holes (6) for bolts (11) and nuts (12) that was used to keep all the plates bolted together and had the optional through-going holes (10) for bolts (9) that were used to keep the bottom plate (7b) and the perforated plate (2) together during the charging of reagents. Furthermore, a thin polymer film (3b) made of Teflon™ was used against the perforations in the perforated plate (2) for tightening. The polymer film had corresponding holes for the bolts used to keep the plates together. Next came the 2 cm thick Teflon™ plate (2) with multiple symmetrically positioned perforations with an 8 mm diameter. In addition, the plate had nine smaller holes (6) going through it for bolts (11) used with nuts (12) to keep all the plates together and had the optional through-going holes (10) for bolts (9) that were used to keep the bottom plate (7b) and the perforated plate (2) together during the charging of reagents. A Teflon™ liner (15) was positioned so that the perforations in the Teflon™ plate were lined with the Teflon™ liner. ~~Another thin, polymer-film (3a) made from 0.5 mm Teflon™ was used in the manner as for (3b).~~ A top plate (7a) was placed above this, identical to the bottom plate (7b), but inverted in relation to it. A frame ensuring good tightening of the outer chambers by preventing lateral deformation of the Teflon™ block (2) may be fastened around the multiautoclave (not shown).

### Example 2

In another embodiment, a multiautoclave may be designed and built as shown in Figure 3. In this case, Teflon™-lined septa (5) type "MICROSEP F138" from Alltech which are stable up to 250°C may be used as bottom- and top-lids for each of the small chambers and individual Teflon™ liners (16) may be used to line each of the perforations.

The multiautoclave would be constructed from the following elements according to Figure 3. A bottom aluminum plate (7b), 2 cm thick with nine smaller, through-going holes (6) for bolts (11) to be used with nuts (12) for holding all the plates bolted together. Above this plate would be mounted a 2 cm thick Teflon™ plate with 100 symmetrically positioned perforations with 8 mm diameters. Further, above and below each perforation would be a 1 mm deep

recess with a 13 mm diameter where Teflon<sup>TM</sup>-lined septa (5) with thickness of 2 mm would be placed. The perforated plate would also have nine smaller, through-going holes (6) for bolts used to keep all the plates bolted together. There is also a set of 6 holes (10) for bolts (9) to hold the bottom plate (7a) and  
5 the central block (2) together. An upper aluminum plate (7b), 2 cm thick, would be placed on top. This plate also has nine smaller through-going holes for bolts (11) to be used with nuts (12) to keep all the plates bolted together.

**CLAIMS:**

1. A pressure and temperature reactor vessel comprising:
  - a) At least a first plate (7a);
  - b) a central block (2) defining a multitude of cavities, the multitude of cavities having at least a first set of open ends;
  - c) a liner (15) associated with the central block positioned so that the liner extends into each of the multitude of cavities (1);
  - d) at least a first seal (3a), operatively associated the plate for engagement with the liner and central block to seal the first set of open ends of the cavities; and
  - e) a locking means (11, 12) acting in concert with the first plate and the central block (2) to engage the seal (3a) and define a multitude of (7a) pressure tight reaction chambers (1).
2. The vessel of Claim 1 further characterized:
  - by a second plate (7b);
  - the central block (2) defining the cavities as a multitude of through-going perforations, the multitude of perforations having the first set of open ends and a second set of open ends at the opposite end of the perforation, with the central block (2) positioned between the first plate (7a) and the second plate (7b);
  - a second seal (3b), operatively associated with the second plate (7b) for engagement with the central block (2) to seal the second set of open ends of the perforations; and
  - the locking means (11, 12) acts in concert with the first and second plates to engage the first and second seals to define the multitude of lined pressure tight reaction chamber.
3. The vessel of Claims 1 or 2 wherein the liner (15) is constructed of a material selected from the group consisting of Teflon<sup>TM</sup>, polyethylene, polypropylene, perfluoroalcoxy fluorinated ethylene propylene, and polyethylethylketone.
4. The vessel of Claims 1 or 2 wherein the liner (15) is from 0.1 to 1 mm in thickness.



5. The vessel of Claims 1 or 2 wherein the central block (2) and the first seal (3a) and any second seal (3b) are individually wholly or partly constructed of a compressible or deformable material.
6. The vessel of Claims 1 or 2 wherein the first plate (7a) and any second plates (7b), acting in concert with the first seal (3a) and any second seal (3b), have a multitude of protrusions (4) of spherical or hemispherical shape, form or profile, either in part or wholly, presenting a curved surface towards the open ends of the multitude of cavities in the central block (2).
7. The vessel of Claims 1 or 2 wherein the first seal (3a) and any second seal (3b) consist of spherical shaped bodies (4) or similar shaped components, fixed to the first and second plates, respectively, such that they present a curved surface towards the open ends of the multitude of cavities in the central block.
8. The vessel of Claims 1 or 2 wherein the first seal (3a) and any second seals (3b) consist of circular disks (5) or other similar circular-shaped components fastened to the first plate (7a) and any second plate (7b), respectively.
9. The vessel of Claim 2 wherein the second seal (7b) consists of a flat plate or membrane, covering all the chambers, fastened to the second plate (7b).
10. The vessel of Claim 2 further comprising a second locking means (9) acting in concert with the second plate (7b) and the central block (2) to further define the multitude of cavities.
11. The vessel of Claims 1 or 2 wherein said first plate (7a), acting in concert with the first seal (3a), has a multitude of protruding profiles having dimensions sufficiently larger than the dimensions of the cavities in the central block (2) and of cross sectional shape such that in concert with the first seal (3a) through the action of the locking means (11, 12), a pressure tight seal is formed over and around each of the multitude of cavities in the central block (2).
12. The vessel of Claims 1 or 2 wherein said central block (2), acting in concert with the first seal (7a), has a multitude of protruding profiles (14) of dimensions sufficiently larger than the dimensions of the cavities and of

cross sectional shape such that in concert with the first seal (3a) and through the action of the locking means (11, 12) on the first plate (7a), a pressure tight seal is formed over and around each of the multitude of cavities in the central block (2).

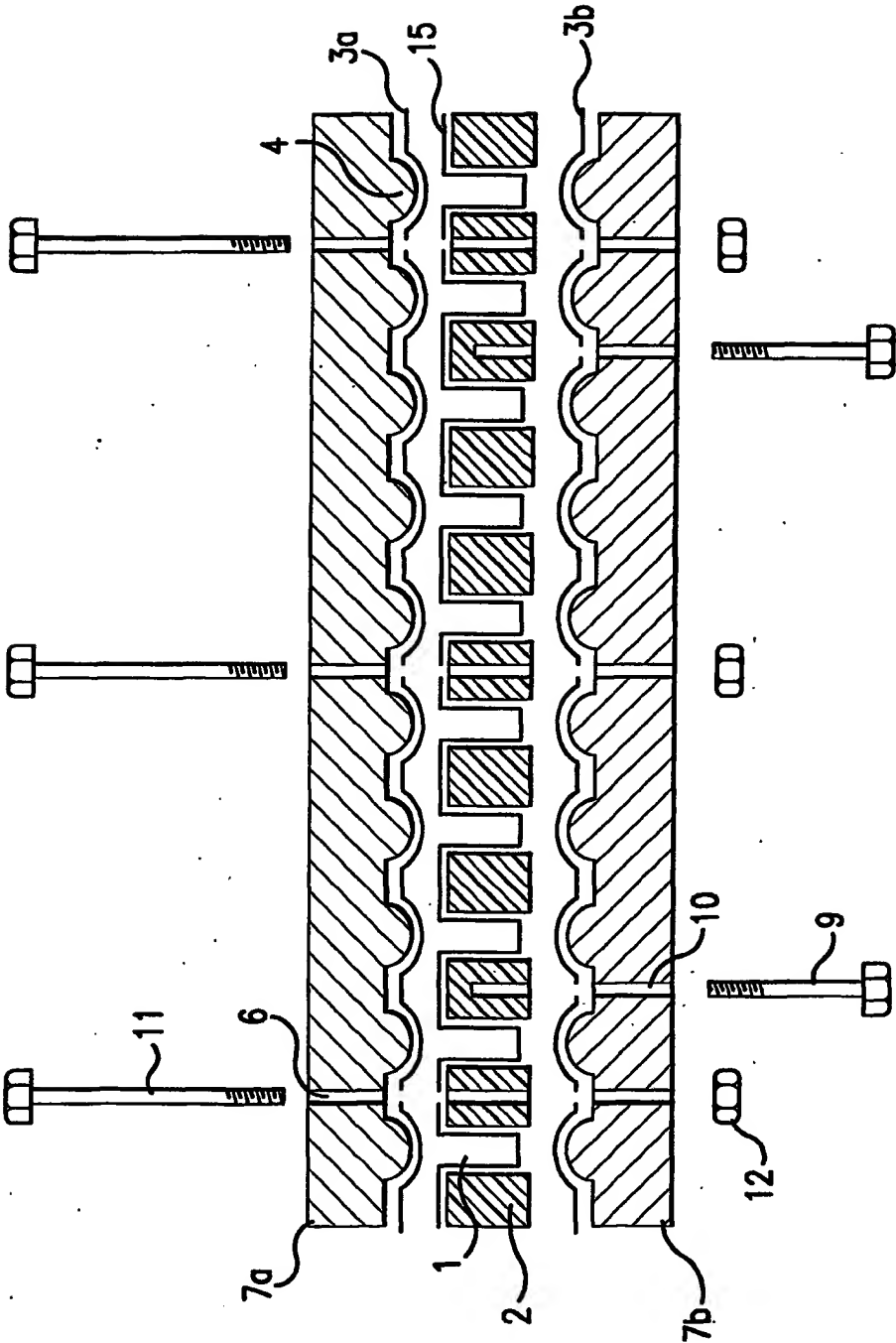


FIG. 1

2/6

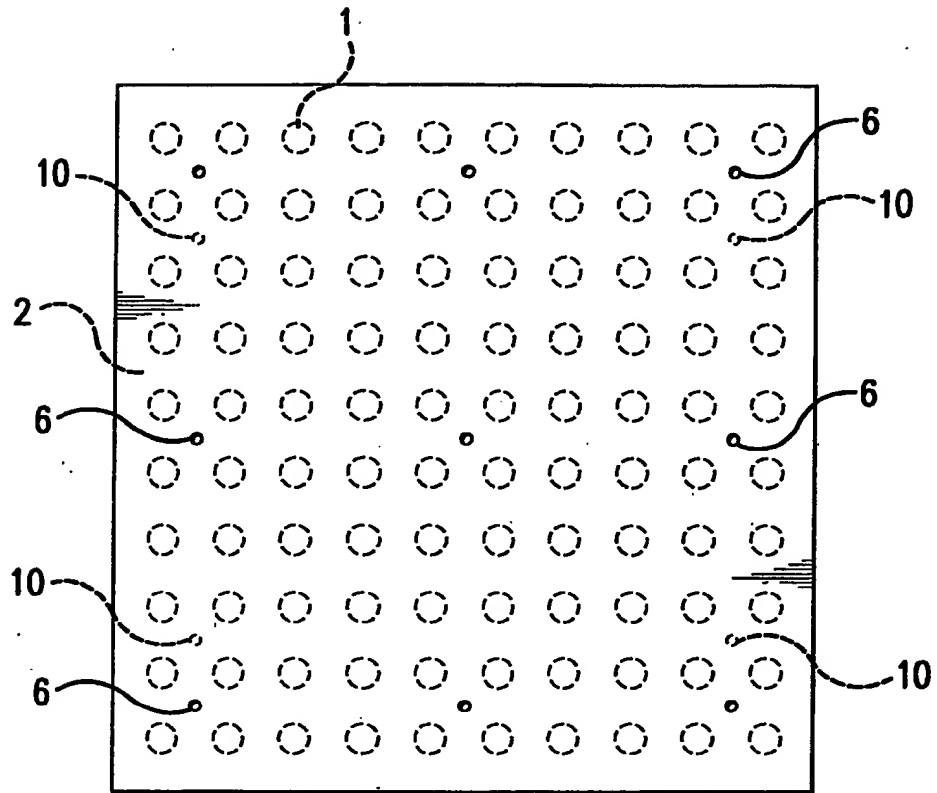


FIG.2

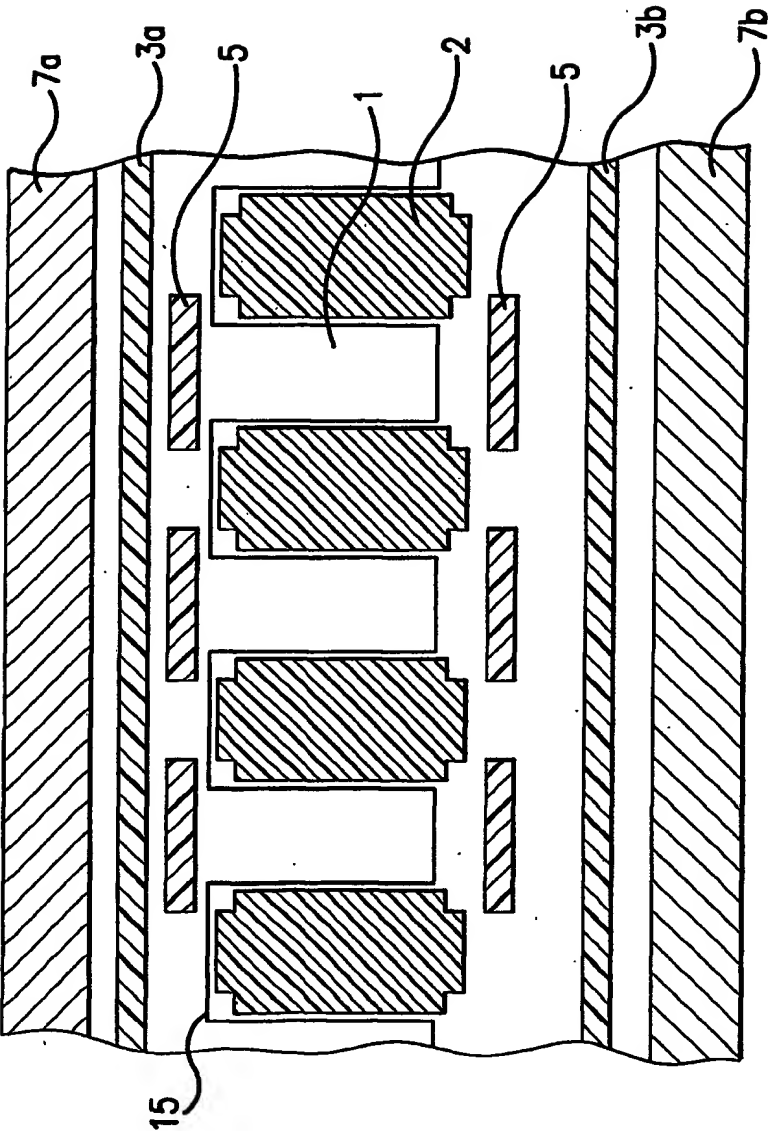


FIG.3

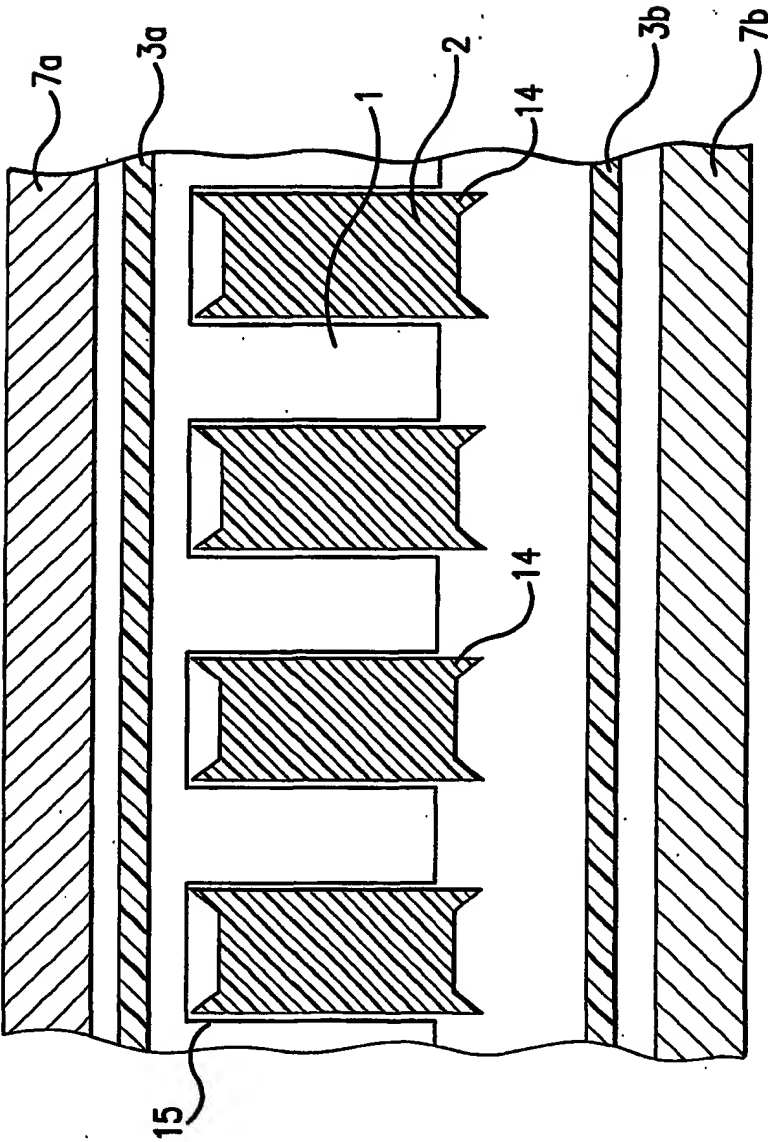


FIG.4

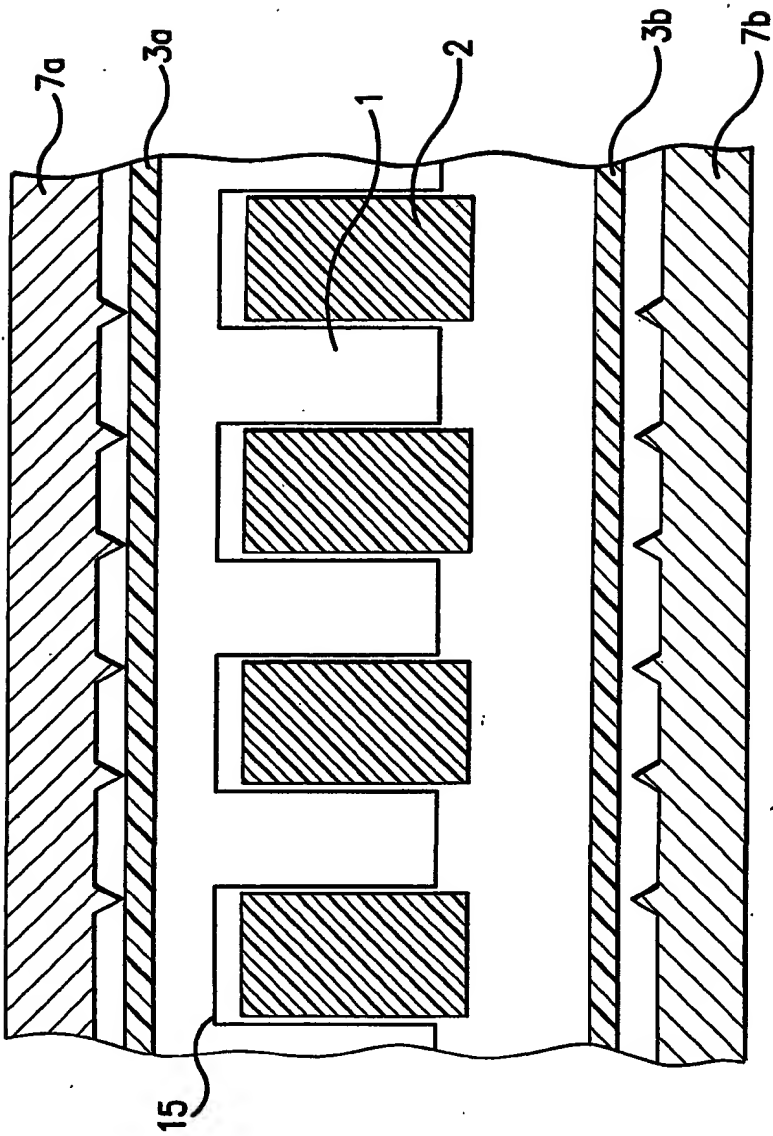


FIG.5

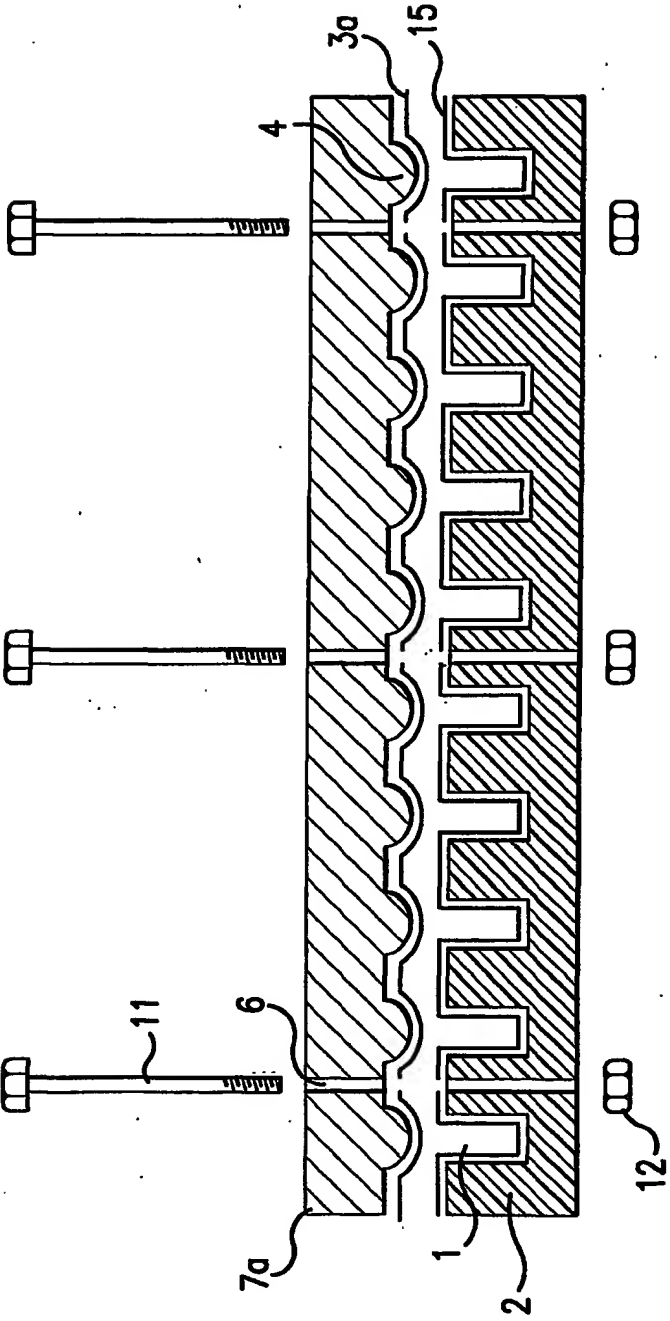


FIG.6